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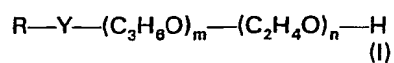
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(54) **Aqueous Dispersions for Use in Textile Dyeing**

(57) Aqueous dispersions of solids, particularly dyestuffs and pigments containing, as dispersing agent, a compound of the formula:



wherein R is a phenyl-phenyl radical; Y is —O—, —CH₂O— or —CO.O—; m+n is from 3 to 30 and n ≥ m.

The dispersions are compatible with most thickening agents, especially synthetic thickening agents, and are therefore of value in the preparation of print pastes for use in the printing of polyester textile materials.

In the formula (I) R may advantageously be 4-phenyl-phenyl.

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SPECIFICATION Aqueous Dispersions

This invention relates to aqueous dispersions of water-insoluble solids, especially pigments and dyestuffs, and to aqueous print pastes derived from the dispersions containing pigments and dyestuffs and especially to print pastes containing "synthetic" thickening agents.

It has been normal practice in the printing of polyester textile materials with disperse dyestuffs to thicken the print pastes with materials of natural origin, such as alginates, but dissatisfaction with their technical performance coupled with a general world shortage of these "natural" thickeners has led to an increase in the use of certain "synthetic" thickening agents, such as polyacrylic acid and its derivatives and homologues, which have recently become available. Although these synthetic thickeners are generally superior to the established natural thickeners in many respects, they have one serious defect in that the viscosity of print pastes made with these thickeners is very sensitive to the presence of ionic species which generally cause a severe depression of viscosity. This defect has caused problems in the use of synthetic thickeners with print pastes containing disperse dyestuffs. These latter are usually dispersed with anionic dispersing agents and the dispersions therefore contain large quantities of ionic species.

The depression of the viscosity of print pastes based on synthetic thickeners when ionic species are added can be overcome to some extent by increasing the quantity of the synthetic thickener but this reduces the commercial acceptability of these thickeners because an important advantage of synthetic thickeners over the natural thickeners is the smaller concentration required, hence the lower cost, to achieve a given viscosity, in the absence of ionic species. A lower level of thickener concentration is also technically desirable because this reduces the amount of washing off required and also reduces interference in the absorption of dyestuffs by the substrate.

In order to accommodate disperse dyestuff dispersions in print pastes containing synthetic thickeners it has been proposed to replace the anionic dispersing agents with non-ionic dispersing agents, see for example UK 1537374. Non-ionic agents have, of course, been used for many years in the stabilisation of pigment dispersions but few of the established non-ionic agents have proved to be satisfactory for the preparation of dispersions of disperse dyestuffs and particularly such dispersions which are designed for incorporation in print pastes based on synthetic thickeners.

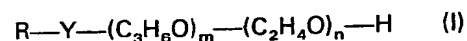
It has been proposed, in Japanese Patent Specification No. 54-30220 (Kokai) to prepare aqueous dispersions of perchlorate salts of basic dyestuffs using poly(ethylene-oxy) derivatives of phenols and naphthols and to use such dispersions in aqueous print pastes containing

65 natural or synthetic thickeners. The print pastes based on synthetic thickeners however contained sufficient thickener (15 to 20%) to have thickened a conventional dispersion of a disperse dyestuff containing an anionic dispersing agent.

70 Furthermore the present applicants have found that the dispersions disclosed in the Japanese specification cannot be used to prepare print pastes of acceptable viscosity unless excessive quantities of thickener are used i.e. from about 5 to 10 times the normal usage.

75 It has now been found that certain aromatic compounds carrying water-solubilising, poly(lower alkylene-oxy) chains are particularly suitable as aids for dispersing water-insoluble inorganic and non-ionic organic solids, particularly pigments and dyestuffs, in aqueous media and that these compounds are especially suitable for preparing dispersions of disperse dyestuffs which will be incorporated into print pastes containing synthetic thickeners. These dispersions are also generally more compatible with natural thickeners than dispersions based on conventional non-ionic dispersing agents. This is an important advantage because printers may use 90 natural and synthetic thickeners for different applications and it is commercially advantageous to be able to use a single colour dispersion in the different applications.

95 According to the present invention there is provided an aqueous dispersion of a finely-divided, water-insoluble solid containing a water-soluble, non-ionic dispersing agent of the formula:



100 wherein R is a phenyl-phenyl radical, Y is $-O-$, $-CH_2O-$ or $-COO-$, $m+n$ is from 3 to 30 and $n \geq m$.

It is preferred that the sum of n and m is from 5 to 25 and more especially from 5 to 15. It is also preferred that $m \leq 5$ and more preferably is zero. It is also preferred that Y is $-O-$.

105 Preferred forms of the groups represented by R are 2-phenyl-phenyl and 4-phenyl-phenyl which may be substituted in either or both rings by one or more non hydrophilic groups such as lower (C_1-C_4) alkyl and halogen, particularly chlorine.

110 Although the dispersions are particularly suitable for use with synthetic thickeners in the preparation of print pastes and so the preferred solids are disperse dyestuffs, the dispersing agents of formula (I) are also suitable for use in the preparation of dispersions of other solids such as pigments, particularly organic pigments, optical brightening agents and anthraquinone, the dispersion of anthraquinone being suitable for use 120 in wood pulping operations for promoting delignification.

As examples of the water-insoluble dyestuffs and pigments which may be incorporated in the present dispersions there are mentioned non-ionic, organic pigments and disperse dyestuffs of the azo, anthraquinone and phthalocyanine series

and inorganic pigments such as metal oxides, iron blues and cadmium yellows.

In addition to the dispersing agent of formula (I) and the water-insoluble solid the dispersion may contain other additives such as anti-microbials, humectants and other surfactants. If the dispersion is to be used in the preparation of a print paste containing a synthetic thickener it is preferred that such additives are non-ionic in order to avoid depression of the viscosity of the print paste.

The dispersion preferably contains from 5% to 70%, by weight of the solid, based on the total weight and from 5% to 150%, by weight of the dispersing agent, based on the weight of the solid, especially preferred ranges being from 10% to 60% and from 20% to 100% respectively.

The mean diameter of the particles of the solid is preferably below 10 microns and more preferably below 5 microns.

The present dispersion may be prepared by any method for the preparation of finely divided dispersions of solids in aqueous media, for example, by grinding the solid together with the dispersing agent in water in a bead or a ball mill until the mean particle diameter of the solid is below 10 microns and more preferably below 5 microns.

Advantages of the present dispersion over other non-ionic dispersions, including those based on closely related dispersing agents, such as the poly(ethylene-oxy) derivatives of alkyl phenols, are greatly reduced foaming during milling, lower viscosity and therefore better handling properties, improved redispersion properties after drying out, improved stability over a range of temperatures and storage periods and greater resistance to "osmotic shock", the disturbance experienced when certain natural thickeners, which are ionic in character, are added to the dispersion during the preparation of a print paste.

Aqueous dispersions of colourants, particularly of disperse dyestuffs, for use in print pastes are generally prepared by the dyestuff manufacturer and may be distributed throughout the world for use by printers in the preparation of print pastes. They are thus frequently stored for long periods under a wide range of climatic conditions in unsealed containers. It is therefore important that the colourant is well protected against flocculation, aggregation and crystal growth so that it remains in a finely divided state until it is applied to a substrate in a print paste. Although precautions are taken to inhibit drying out, by the use of humectants, this cannot be completely avoided and it is therefore important that the dried dispersion should be readily redispersible by a simple mixing procedure without the formation of lumps and specks which can spoil print quality. Finally it is important that the dispersion should not be sensitive to dilution and the addition of the ingredients which are conventionally used in the preparation of aqueous print pastes.

As indicated hereinbefore the dispersions of the present invention, wherein the solid is a

colourant, i.e. a pigment or a dyestuff, are particularly suitable for the preparation of print pastes and according to a further feature of the present invention there is provided an aqueous print paste comprising a finely-divided water-insoluble inorganic or non-ionic organic colourant, a dispersing agent of formula (I) and a thickening agent.

The term thickening agent includes both natural and synthetic thickening agents although preferred print pastes are those containing a disperse dyestuff and a synthetic thickening agent. The term "synthetic" thickening agent includes all those agents, suitable for the thickening of print pastes, which are not derived from natural products and, as examples there may be mentioned polyacrylates, such as polyacrylic acid and its derivatives and homologues, polyvinyl alcohol and ethylene/maleic anhydride copolymers. As examples of natural thickening agents there may be mentioned locust bean/mannogalactan gum, guar gum (mannogalactan), sodium alginates, polysaccharides, starch ethers, hydroxylated and carboxylated starch ethers and oil-in-water emulsions.

A print paste based on a natural thickening agent preferably contains from 4% to 5% of the thickening agent whereas one based on a synthetic thickening agent preferably contains from 0.5% to 1.5% of the thickening agent. The weight of colourant may vary within wide limits depending on the depth of shade of print required and preferred proportions of the dispersing agent, in relation to the colourant, are those indicated hereinbefore in respect of the dispersions.

The print paste may be prepared in any convenient manner, a preferred method in the case of a disperse dyestuff dispersion being to dilute the dispersion with water and to add a concentrated solution of the thickening agent with vigorous agitation. Any other ingredients, such as wetting agents, fixation promoters and preservatives, may then be added to the paste.

The invention is further illustrated by the following Examples. Unless otherwise stated, all parts and percentages are by weight.

Example 1

19.2 Parts of the dispersing agent of formula (I) wherein R is 2-phenyl-phenyl, Y is —O— , m is 0 and n is 10, 15.4 parts of dipropylene glycol and 1.2 parts of a 20% aqueous solution of sodium 2-phenylphenate are dissolved in 35.8 parts of water and the pH is adjusted from 10 to 7 with a 10% aqueous solution of acetic acid.

48.4 Parts of a press paste of the dyestuff, 2-acetyl-amino-4-[N,N-di(β -methoxycarbonyl-ethyl)amino]-4'-nitroazobenzene, containing 24 parts of dyestuff and 24.4 parts of water is added to the above solution and homogenised for 10 minutes with a higher shear (Silverson) mixer. The dyestuff slurry is then stirred in a stainless steel, water-cooled vessel with 360 parts of 0.3 to 0.5 diameter glass beads. Agitation is effected by

three axially-aligned, 60 mm diameter nylon discs mounted 25 mm apart on a stainless steel shaft rotating at 3000 revolutions per minute. After 30 minutes agitation the mean particle diameter of the dyestuff is below 2 microns and the dispersion is deflocculated.

The dispersion, containing 20% dyestuff and 16% dispersing agent is separated from the glass beads by filtration through a nylon cloth having 25 micron pores.

Example 2

The procedure of Example 1 is repeated using 15.4 parts of the condensate of glycerol with 4 molecular equivalent of propylene oxide (Daltolac 90) in place of the 15.4 parts of dipropylene glycol.

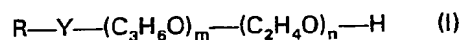
Example 3

The procedure of Example 1 is repeated using 2.4 parts of a lignosulphonate (Reax 85A) in place of the 15.4 parts of dipropylene glycol and increasing the quantity of water to 48.4 parts.

The dispersions of Examples 1 to 3 all give commercially acceptable prints on polyester textile material when incorporated into print pastes based on natural or synthetic thickening agents.

Claims

1. An aqueous dispersion of a finely-divided water-insoluble solid containing a water-soluble, non-ionic dispersing agent of the formula:



wherein R is a phenyl-phenyl radical; Y is $-O-$, $-CH_2O-$ or $-COO-$; $m+n$ is from 3 to 30 and $n \geq m$.

2. An aqueous dispersion according to Claim 1 wherein $n+m$ is from 5 to 15.
3. An aqueous dispersion according to Claim 1 or Claim 2 wherein m is from 0 to 5.
4. A dispersion according to any one of Claims 1 to 3 wherein R is 4-phenyl-phenyl.
5. A dispersion according to any one of Claims 1 to 4 wherein the solid is a disperse dyestuff or a pigment.
6. A print paste comprising a dispersion according to any one of Claims 1 to 5 and a thickening agent.
7. A print paste according to Claim 6 wherein the thickening agent is synthetic.
8. An aqueous dispersion of a disperse dyestuff as described in any one of the Examples.